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LEAD ELECTROWINNING FROM AQUEOUS CHLORIDE ELECTROLYTE

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by

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ABSTRACT

A laboratory diaphragm cell featuring a cathode compartment with a tapered bottom has been developed, and experimental conditions have been established for electrowinning loose, uniform, leaf-like crystals of lead from aqueous chloride electrolyte. The lead deposit characteristics were relatively insensitive to variations in the applied current density, cathode material, cathode rotation speed and to glue additions in the electrolyte. Lead electrowinning from aqueous chloride electrolyte was not adversely affected by the presence of sulphate ion (added as $PbSO_4$), but both bismuth and copper co-deposited with the lead. Hot water was effective in displacing entrained chloride salts resulting in a final product having a purity of 99.98% Pb.

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ELECTRO EXTRACTION DU PLOMB DE L'ÉLECTROLYTE DE CHLORURE AQUEUX

par

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RÉSUMÉ

Une cellule de diaphragme de laboratoire entourée d'une gaine à base fuselée a été mise au point et des conditions expérimentales ont été établies pour l'électro extraction de cristaux de plomb libres, uniformes, en lames minces, de l'électrolyte de chlorure aqueux. Les caractéristiques du précipité de plomb ont été relativement insensibles aux variations de la densité du courant appliqué, au matériau de la cathode, au régime de rotation et aux ajouts de colle à l'électrolyte. Le processus d'électro extraction du plomb de l'électrolyte de chlorure aqueux n'a pas été affecté de façon négative par la présence de l'ion de sulfate (ajouté sous forme de $PbSO_4$), mais le bismuth et le cuivre se sont simultanément déposés avec le plomb. De l'eau chaude a réussi à déplacer les sels de chlorure entraînés, ce qui a permis d'obtenir un produit fini d'une pureté de 99,98 % Pb.

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INTRODUCTION

Lead electrodeposits obtained from aqueous chloride electrolyte are characteristically non-compact and powdery, even when produced at low current densities. Attempts to produce smooth, dense, compact deposits of lead from such systems have met with only limited success (1-4), and as a result, some workers have opted to electrowin lead as a metallic powder.

Haver et al, (5) studied various electrolytes including spent ($FeCl_2$-NaCl) leach solution, HCl, NaCl and NH_4Cl saturated with $PbCl_2$. In all tests, 35g of $PbCl_2$ were placed in the cell, and formed a layer about 3 mm thick over the horizontal cathode. After the addition of 800 mL of the desired electrolyte, electrolysis was started using controlled bath composition, temperature, current density and electrode spacing. During electrolysis, $PbCl_2$ covering the cathode is converted to metallic lead powder and either ferric ion or chlorine gas is formed at the anode depending on the electrolyte employed. The metallic lead powder is not attached to the cathode and can be "raked" out of the cell, washed and melted to obtain a high purity lead product.

Hazen Research has developed a conceptual design for a bipolar aqueous lead chloride electrolysis cell (6). The design incorporates sloping anode-cathode pairs such that $PbCl_2$ crystals, resulting from the saturated $PbCl_2$ electrolyte, reside on the sloping cathode surface. The electrolyte is 5% NaCl at 60°C and the operating current density is 150 ASF (1605 A/m^2). Feed slurry containing 10% $PbCl_2$ solids is delivered to the cell at a rate of 53 L/min. The cell discharges a slurry containing 7% $PbCl_2$ + 3% metallic Pb. After gravity separation, the $PbCl_2$ crystals are recycled to the cell.

Minemet Recherche (7) is developing a ferric chloride-brine leach-electrowin process for treating galena concentrates. The purified

pregnant leach solution is fed to the cathode compartment of a diaphragm electrolysis cell. The solution is maintained at 85°C and contains 29.7 g/L Pb, 250 g/L NaCl and 10 g/L excess ferrous ion to ensure that chlorine is not formed at the anode. The cathode compartment features a tapered bottom connected to a storage vessel used to collect the lead powder which falls from the cathode. The catholyte is circulated by means of a pump between the storage vessel and the cathode compartment to help dislodge the lead particles.

Duval Corporation has also done some work to develop a design for an aqueous lead chloride electrolysis cell (8). The $PbCl_2$ crystals were dissolved in 4 M NaCl solution to prepare the cell electrolyte. Duval used a copper cathode covered with insulating paint; holes were exposed in the paint to provide high current density sites for lead metal deposition. The cells employed graphite anodes and were constructed of FRP. A tightly woven cloth membrane was used to separate the cathode and anode compartments. The lead crystals formed on the cathode fell either naturally or by imposed impact, and dropped to the bottom of the cell. They were collected on a moving rubber belt passing along the bottom of the cell. The coarse-grained lead crystals were collected from the belt, washed and then melted.

The electrowinning of lead as a loose powder rather than as a compact deposit offers the advantage of operating the cell in a continuous manner at a high current density. Varying the current density in the presence of certain additives in the electrolyte may permit the production of uniform lead crystals which are easier to handle and less prone to oxidation than fine powder. Thus, the present work is concerned with developing a cell and experimental conditions whereby uniform, loosely held crystals are formed at the cathode.

EXPERIMENTAL

The electrolysis cell and electrolyte circulation system are shown schematically in Figure 1. The circular cell with conical bottom was constructed from a 0.64 cm wall acrylic cylinder having an inside diameter of 10.8 cm. The 12.1 cm high acrylic cylinder was joined to a 5.1 cm thick, 12.7 cm diameter acrylic base. The tapered cell bottom was milled into the acrylic base prior to cell assembly. The conical bottom of the cell was linked to a collector flask via a spigot. The anode compartments were separated from the cathode compartment by Dynel cloth diaphragms which were fixed to the bottom and to the walls of the cell. The distance between the diaphragms was 6.4 cm. The cell cover contained two slots to accommodate the DSA anodes which were fixed at the bottom of the anode compartments. The cover also contained a 1.3 cm diameter hole in the center to accommodate a rotating cylinder cathode.

The electrolyte was prepared using reagent grade chloride chemicals. Unless otherwise stated, the cell electrolyte contained 10 g/L $PbCl_2$, 3.5 M NaCl and 1.0 M HCl. The feed electrolyte had the same composition as the cell electrolyte except that it contained 20 g/L $PbCl_2$. Glue was added to both the cell and feed electrolytes as required from a separate stock solution. The total volume of cell electrolyte was 4 L: 2 L in the reservoir and 1 L in each of the cell and collector flask. The 4 L of solution was continuously circulated through the cell, collector flask and reservoir by means of a pump. The level of the catholyte was maintained about 1 cm higher than the anolyte levels to ensure a flow of spent catholyte through the diaphragms into the anode compartments. Spent anolyte was pumped from the anode compartments and was collected in a separate tank. The cell reservoir was sealed so that the vacumm created by the circulating electro-

lyte allowed feed electrolyte to flow into the system to replace the spent anolyte. The rate of addition of the feed electrolyte was controlled by a glass-Teflon fine metering valve.

During electrolysis, lead deposited as loosely adherent crystals on the rotating cathode. The turbulence caused by the rotating cathode and circulating electrolyte dislodged the lead crystals which fell through the tapered cell bottom into the collector flask. At the end of the electrolysis period, usually 1-h, the lead was removed from the collector flask and was washed several times with hot water to displace entrained chloride salts.

RESULTS AND DISCUSSION

1. Effect of Current Density

The effect of current density on lead electrowinning from aqueous chloride electrolyte was studied using a rotating Ti rod cathode. The immersed surface area of the electrode was 0.0013 m^2 (0.014 ft^2) and it was rotated at 1500 RPM. The composition of the cell electrolyte was 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl and it was maintained at 70°C. The feed solution consisted of 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl heated to ~ 90°C to maintain $PbCl_2$ solubility. The feed electrolyte was bled into the circulating cell electrolyte at a rate sufficient to maintain the $PbCl_2$ concentration at about 10 g/L. The current efficiency (CE), energy requirement (ER) and deposit characteristics obtained at the various current densities are summarized in Table 1. The CE reached a maximum value of 99.1% at a current density of 2310 A/m^2 (210 A/ft^2); the corresponding ER was 1.2 kW/kg. At 3300 A/m^2 (300 A/ft^2), the CE decreased to 82%, probably because of too rapid a depletion of $Pb^{}$ ion from the diffusion layer.

The lead deposit was very similar for all values of the current density. The lead formed on the cathode as flat, leaf-like platelets which

grew vertically into the solution." After a short time, clusters of these platelets separated from the cathode and fell to the bottom of the cell and subsequently into the collector flask." A typical lead deposit is shown in the photomicrograph in Figure 2."

When the cell was operated at a lower $PbCl_2$ concentration; i.e., 5 g/L, the optimum CE occurred at 1650 A/m^2 (150 A/ft^2), Table 2. On the other hand, increasing the cell electrolyte composition to 15 g/L $PbCl_2$ increased the CE at 3300 A/m^2 to 94.7%, Table 2."

2. Effect of Cathode Material

The effect of cathode material on lead electrowinning from an electrolyte containing 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl was studied at various current densities." The cathodes were prepared from Ti, graphite and stainless steel rods and were rotated at 1500 RPM. The results are summarized in Table 3." As indicated, the CE obtained using both Ti and graphite cathodes averaged ~ 96% whereas the CE obtained using the stainless steel cathode averaged only 90%." The lead deposit was essentially the same as described in the previous section; significant differences were not observed for the various cathode materials.

3. Effect of Glue

A previous study [4] showed that glue additions < 100 mg/L were not effective in eliminating dendritic lead growth." Thus, in the present study, the effect of glue concentrations in the range 150 to 900 mg/L on the formation of crystalline lead deposits was determined. The effect of glue was studied using a graphite cathode rotated at 1500 RPM for current densities of 990 and 1650 A/m^2 (90 and 150 A/ft^2). The results obtained for a solution containing 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl at 70°C are summarized in Table 4. Glue additions to 900 mg/L did not produce any

outstanding changes in the lead deposit characteristics; at high glue concentration the lead platelets were more distinctive than at glue concentrations lower than 600 mg/L. At 900 A/m^2, the addition of glue resulted in a consistently lower CE; i.e., ~ 93% rather than 97% in the absence of glue.

4. Effect of Cathode Rotation Speed

The effect of cathode rotation speed on lead electrowinning was studied using a graphite cathode at a current density of 990 A/m^2. At low stirring speeds, < 800 RPM, the lead platelets tended to grow into larger clusters before separating from the cathode. The results are summarized in Table 5.

5. The Effect of Lead Sulphate

Sulphate ion is known to have an adverse effect on lead electrowinning from lead chloride during molten salt electrolytes. Thus, Fleck et al. (9) observed that when the sulphate concentration increased from 1 to 3%, the CE decreased from 98 to 93% and the voltage increased from 2.5 to 2.8 V. They also noted that a salt crust formed on the surface of the electrolyte when the sulphate increased to > 2% because the sulphate solubility in the chloride melt was exceeded.

The effect of $PbSO_4$ on lead electrowinning from aqueous chloride electrolyte was studied by replacing a known percentage of the $PbCl_2$ with $PbSO_4$ in both the cell and the feed electrolytes. The effect of $PbSO_4$ was determined at 990 A/m^2 using a graphite cathode rotated at 1500 RPM. The results obtained are summarized in Table 6. As the data indicate, the replacement of up to 25% of the $PbCl_2$ feed in the electrolyte by $PbSO_4$ had no significant effect on the CE, ER or lead deposit characteristics.

6. Analysis of the Lead Deposits

At the end of the electrolysis period, the collector flask was detached from the cell and the lead product was recovered by vacumm filtration. The lead product was washed several times with hot water to remove entrained electrolyte. Selected samples were analyzed for total Cl^-, total Na^+ and, where appropriate, for total SO_4^{2-}. The results are summarized in Table 7. Most of the samples analysed had a total Cl^- content of $\sim$ 0.01% (100 ppm); lead electrowon in the presence of SO_4^{2-} contained < 0.005% (< 50 ppm) SO_4^{2-}. In terms of total Cl^-, Na^+ and SO_4^{2-} content, the lead purity was 99.98% as compared to 99.94% for corroding lead.

7. Effect of impurities

Some preliminary tests were conducted on the effect of Bi, Cd and Cu on lead electrowinning from aqueous chloride electrolyte. The tests were done at 990 A/m^2 using a Ti cathode rotated at 1500 RPM. The impurities were added as chloride salts to an electrolyte containing 10 g/L $PbCl_2$+3.5 M NaCl + 1.0 M HCl at 70°C. After the 1-h electrolysis period, the lead product was recovered, washed and analysed for the impurity metal using AA spectrophotometry. The results are summarized in Table 8 and indicate that the presence of 10 mg/L Cu is very detrimental to aqueous lead electrowinning. Bismuth also co-deposits with lead to a significant extent but Cd, which is less noble than Pb, does not co-deposit significantly under these experimental conditions.

8. Electrolysis Containing $FeCl_2.4H_2O$

Several tests were done using a cell electrolyte containing 0.8 M $FeCl_2.4\,H_2O$ + 2.3 M HCl + 1.0 M HCl and 10 g/L $PbCl_2$ at 70°C. Such an electrolyte is representative of that which would result from the ferric chloride leaching of a galena concentrate. The results obtained

using a graphite rod cathode rotated at 1500 RPM are summarized in Table 9. The deposit characteristics were the same as those obtained from the electrolytes which did not contain $FeCl_2$, $4H_2O$, Glue, which did not significantly change the deposit characteristics, resulted in a decrease in CE. The ER values are decreased in the presence of $FeCl_2$, $4H_2O$ because the anodic reaction is the oxidation of Fe^{2+} to Fe^{3+} rather than Cl^- to Cl_2.

CONCLUSIONS

A laboratory cell has been designed and experimental conditions have been established for electrowinning uniform lead crystals from aqueous chloride electrolyte. A circular diaphragm cell featuring a cathode compartment with a tapered bottom facilitates the recovery of the lead crystals which are formed on a rotating cylinder cathode. The characteristics of the lead crystals are relatively insensitive to the applied current density, glue addition, cathode rotational speed and cathode material. Higher lead concentrations in the cell electrolyte allow higher current densities to be achieved before the on-set of hydrogen evolution. The replacement of up to 25% of the $PbCl_2$ in the feed electrolyte by $PbSO_4$ did not have an adverse effect on lead electrowinning from aqueous chloride electrolyte. Preliminary tests on the effect of impurities on lead electrowinning indicated that both Cu and Bi co-deposited with Pb; Cd did not co-deposit with Pb to a significant extent. Washing the Pb product with hot water proved to be effective for removing entrained Cl^-, $SO_4^=$ and Na^+ ions. Analysis of selected lead deposits indicated that the product was 99.98% Pb.

ACKNOWLEDGEMENT

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Table 1. The Effect of Current Density on Lead Electrowinning from Aqueous Chloride Electrolyte

Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
990	100	1,0	black, shiny, metallic, flat, leaf-like platelets
1320	96,6	1,0	black, shiny, metallic, flat, leaf-like platelets
1650	95,7	1,3	black, shiny, metallic, flat, leaf-like platelets
1980	95,1	1,4	gray platelets; not as distinctive
2310	99,1	1,2	gray, metallic platelets;
2640	97,1	1,4	gray, well formed platelets
3300	82,0	1,7	gray, metallic platelets; smaller in size

Ti cathode (0,0013 m^2); 1500 RPM
Cell Electrolyte: 10g/L $PbCl_2$+3,5 M NaCl + 1,0 M HCl at 70°C
Feed Electrolyte: 20g/L $PbCl_2$+3,5 M NaCl + 1,0 M HCl at 90°C

Table 2. Effect of $PbCl_2$ Concentration on Lead Electrowinning from Aqueous Chloride Electrolyte

$PbCl_2$ (g/l)	Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
5	990	94,0	0,98	metallic, shiny, leaf-like platelets
5	1650	97,0	1,1	metallic, shiny, leaf-like platelets
5	1980	92,0	1,3	gray, dull, platelets;
5	3300	40,0	3,7	yellow powder
15	3300	94,7	1,4	gray, metallic, platelets
15	3630	92,4	1,5	gray, metallic, coarse platelets

Ti cathode (0,0013 m^2); 1500 RPM

Table 3. Effect of Cathode Material on Lead Electrowinning from Aqueous Chloride Electrolyte

Cathode Material	Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
Ti	1320	96.6	1.0	black, shiny, metallic, flat platelets
Ti	1650	95.7	1.3	black, shiny, metallic, flat platelets
Graphite	990	97.4	0.9	dull gray platelets
Graphite	1650	96.2	1.1	dull gray platelets
Stainless steel	1320	91.0	1.1	shiny gray platelets
Stainless steel	1650	89.7	1.3	dull gray platelets

Cathodes rotated at 1500 RPM
Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl at 70^oC
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl at 90^oC

Table 4. Effect of Glue on Lead Electrowinning from Aqueous Chloride Electrolyte

Glue (mg/L)	Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
0	990	97.4	0.90	dull gray leaf-like platelets
0	1650	96.2	1.1	dull gray leaf-like platelets
150	990	92.8	0.95	dull gray leaf-like platelets
150	1650	97.9	1.2	dull gray leaf-like platelets
300	990	92.6	1.0	dull gray leaf-like platelets
300	1650	96.1	1.3	gray, uniform platelets
600	990	95.1	0.90	platelets more distinctive
600	1650	90.4	1.3	smaller platelets; clusters
900	990	92.7	0.98	platelets more pronounced
900	1650	95.3	1.3	platelets; some finer crystals

Graphite Cathode (0.0023 m^2); 1500 RPM
Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl; 70°C
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl; 90°C

Table 5. Effect of Cathode Rotation Speed on Lead Electrowinning from Aqueous Chloride Electrolyte

Rotation Speed (RPM)	Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
0	990	96.0	1.1	small, flat, leaf-like platelets
200	990	98.6	0.97	small, metallic clusters of platelets
500	990	96.2	0.99	small, metallic clusters of platelets
800	990	98.6	0.97	metallic platelets, not as clustered
1200	990	100.0	0.94	metallic platelets, not as clustered
1500	990	97.4	0.90	dull, gray, platelets

Graphite Cathode (0.0023 m^2)
Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl

Table 6. Effect of $PbSO_4$ on Lead Electrowinning from Aqueous Chloride Electrolyte.

$PbSO_4$ (% of $PbCl_2$)	Current Density (A/m^2)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
0	990	97.4	0.98	dull gray, leaf-like platelets
5	990	96.0	0.94	fine, platelets; clusters
10	990	97.6	1.0	gray, metallic platelets
15	990	95.7	1.0	gray, metallic platelets
20	990	96.0	0.94	gray, metallic platelets
25	990	94.7	0.99	gray, metallic platelets

Graphite Cathode (0.0023 m^2), 1500 RPM
Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl

Table 7. Analysis of Lead Deposits obtained under Various Electrolysis Conditions of Total Cl, Na and SO_4^{2-}

Current Density (A/m^2)	Glue (mg/L)	Rotational Speed (RPM)	Cathode Material	$PbSO_4$ (% of $PbCl_2$)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Total Cl^- (%)	Total Na^+ (%)	Total SO_4^{2-} (%)
1650	900	0	Ti	0	96.4	0.80	0.025	0.0078	--
990	0	1500	Graphite	0	95.5	0.90	0.007	0.0039	--
1980	0	1500	Graphite	0	93.4	1.1	0.010	0.0070	--
990	900	1500	Graphite	0	92.0	1.0	0.007	0.0051	--
990	0	200	Graphite	0	97.6	1.1	0.009	0.0065	--
990	0	500	Graphite	0	98.6	0.96	0.011	0.0054	--
990	0	1500	Ti	0	96.1	0.97	0.011	0.0049	--
990	0	1500	SS	0	94.5	1.2	0.010	0.0053	--
990	0	1500	Graphite	5	96.0	0.97	0.011	0.0053	< 0.005
990	0	1500	Graphite	25	94.7	0.98	0.011	0.0049	< 0.005

Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl at 70°C
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl at 90°C

Table 8. The Effect of Certain Impurities on the Quality of Lead Deposits Electrowon from Aqueous Chloride Electrolyte

	Percent Impurity in Lead Deposit	
Concentration (g/L)	0,01	0,05
Impurity		
Bi	1,2	3,4
Cd	< 0,002	< 0,002
Cu	29,2	-

Ti Cathode (0.0013 m^2); 1500 RPM; 990 A/m^2
Cell Electrolyte: 10 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl
Feed Electrolyte: 20 g/L $PbCl_2$ + 3.5 M NaCl + 1.0 M HCl

Table 9. Effect of Current Density and Glue on Lead Electrowinning from Aqueous Chloride Electrolyte Containing 0.8 M $FeCl_2.4H_2O$

Current Density (A/m^2)	Glue (mg/L)	Current Efficiency (%)	Energy Requirement (kWh/kg)	Deposit Characteristics
1650	300	91.0	0.88	metallic, shiny, leaf-like platelets
1650	600	92.0	0.89	metallic, shiny, leaf-like platelets
1650	0	100.0	0.81	dull gray, uniform, leaf-like platelets
2970	0	100.0	1.0	dull gray uniform flat platelets
1650	50	100.0	0.73	metallic, leaf-like platelets
1650	1200	94.0	0.91	dull gray, uniform, leaf-like platelets
2970	1200	93.0	1.1	dull gray, leaf-like platelets

Graphite Cathode (0.0023 m^2); 1500 RPM
Cell Electrolyte; 0.8 M $FeCl_2 4H_2O$ + 2.3 M NaCl + 1.0 M HCl + 10 g/L $PbCl_2$ at 70°C
Feed Electrolyte: 0.8 M $FeCl_2.4H_2O$ + 2.3 M NaCl + 1.0 M HCl + 20 g/L $PbCl_2$ at 90°C

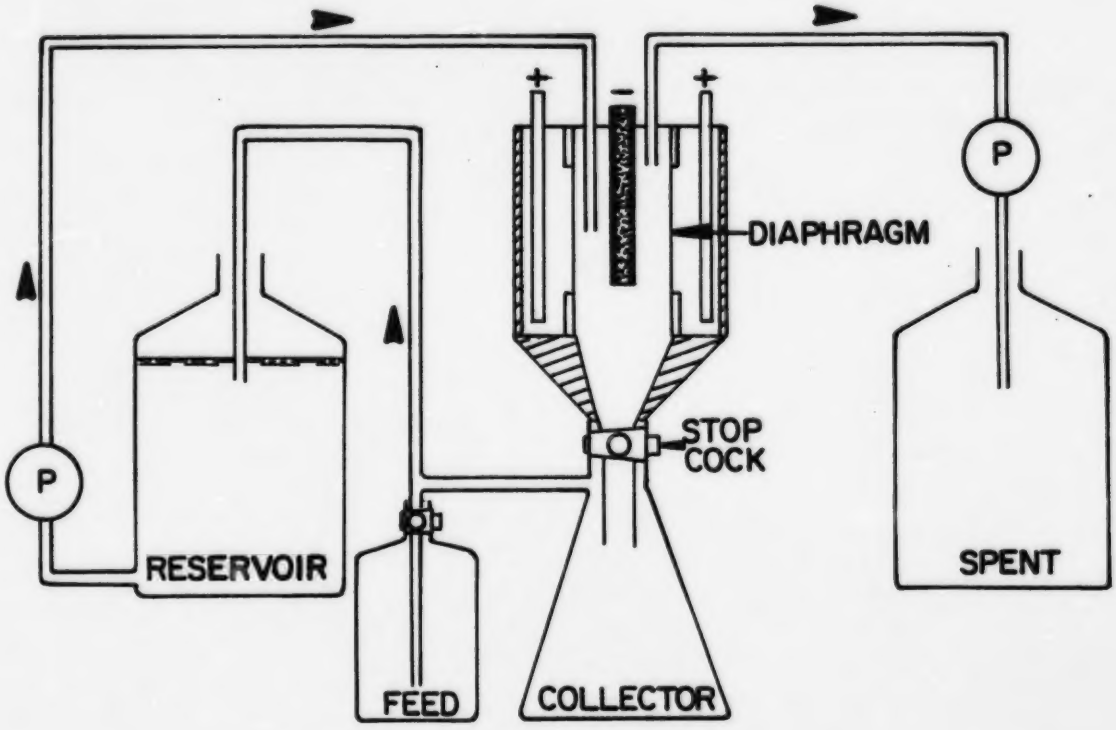

Figure 1. Electrolysis Cell and Electrolyte Circulation System.

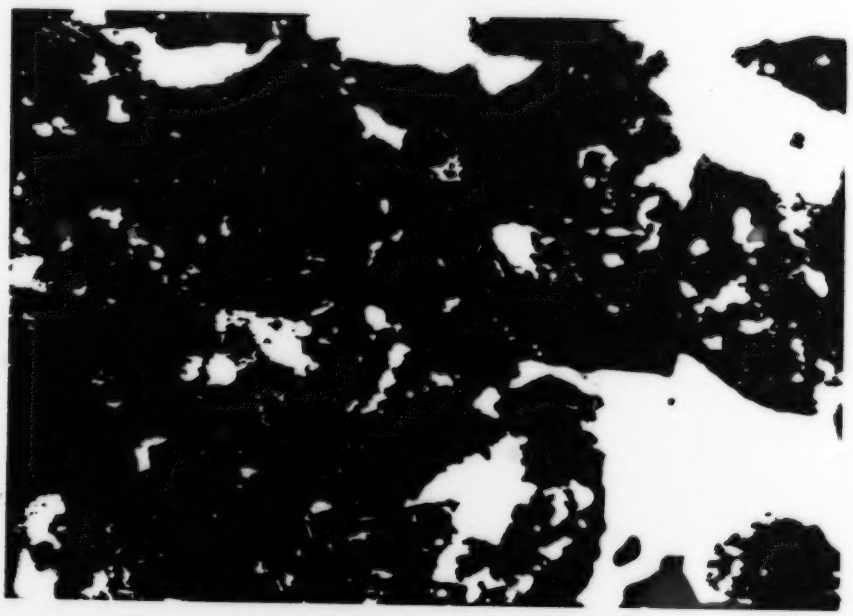

Figure 2. Photomicrograph (X135) of lead crystals